

Pf-2727/ncc/us/mh

## METHOD OF PROCESSING A NANOTUBE

5

## BACKGROUND OF THE INVENTION

The present invention relates to a method of processing a nanotube, and more particularly to a method of processing a nanotube, which is suitable for cutting the nanotube and for forming a top of the nanotube.

A single-layer winded carbon nanotube has an extremely fine structure in nanometer order. Properties of the single-layer winded carbon nanotube have been on the investigation. The research and developments of the single-layer winded carbon nanotube for application have been active. The single-layer winded carbon nanotube comprises a cylindrically winded single layer of graphite hexagonal network. An electron structure largely varies depending upon a diameter of the tube and a chiral angle, for which reason the electrical conductivity of the carbon nanotube varies between a conductivity of a metal and a conductivity of a semiconductor, and further the carbon nanotube exhibits a property similar to one-dimensional electric conduction.

The carbon nanotube may be applied to a field emitter. This field emitter has been known and is disclosed in (1) W.A. de Heer, A.Chatelain,

Pf-2727/nec/us/mh

- and D. Ugarte, Science 270, 1179 (1995) ; (2) A.G. Rinzler, J.H. Hafner, P. Nikolaev, L. Lou, S.G. Kim, D. Tomanek, P. Nordlander, D.T. Colbert, and R.E. Smalley, Science, 269, 1550 (1995) ; (3) P.G. Collins and A. Zettl, Appl. Phys. Lett., 69, 1969 (1996) ; (4) Q.H. Wang, T.D. Corrigan, J.Y. Dai, 5 P. R. H. Chang, and A.R. Krauss, Appl. Phys. Lett., 70, 3308 (1997) ; (5) Y. Saito, K. Hamaguchi, T. Nishino, K. hata, K. Tohji, A.Kasuya, and Y. Nishina, Jpn. J. Appl. Phys., 36, L1340 (1997) ; (6) J-M. Bonard, J-P. Salvetat, T. Stockli, W.A. de Heer, L. Forro, and A. Chatelain, Appl. Phys. Lett., 73, 918 (1998).
- 10           The sharpness of the top of the field emitter of the carbon nanotube emphasizes the field effect and realizes the good characteristic of the field emission.
- No practicable method of operating the nanotube tip and controlling the same has been established, for which reason the 15 conventional carbon nanotube is extremely low in probability of having an optimum directionality and serving as an useful electron emitter. As shown in (7) Y. Zhang and S. Iijima, Philos. Mag. Lett., 78, 139 (1998), the most of the manufactured single layer winded carbon nanotube is large in aspect ratio or is slender and further is curved, for which reason it is difficult to 20 practice the single layer winded carbon nanotube.

It is actually difficult that the plural nanotube tips are arranged in the same direction and the plural nanotubes are aligned along a single line. As shown in the above literature (7), it is actually difficult to evaluate the nanotube tip with a probe.

Pf-2727/nec/us/mh

In order to have solved the above problem, there was proposed a method of cutting the single carbon nanotube by an oxidation using a nitric acid, an acid mixing with a nitric acid or a sulfuric acid. The cutting method is disclosed in (8) K.B. Shelimov, R.O. Esenaliev, A.G. Rinzler, C.B. Huffman, and R.E. Smalley, Chem. Phys. Lett., 282, 429 (1998) ; (9) J. Liu, M. J. Casavant, M.Cox, D.A. Walters, P. Boul, W. Lu, A.J. Rimberg, K.A. Smith, D.T. Colbert, and R.E. Smalley, Chem. Phys. Lett., 303, 125 (1999) ; (10) Z. Shi, Y. Lian, F. Liao, X. Zhou, Z. Gu, Y. Zhang, and S. Iijima, Solid State Comm., 112 (1999) 35.

10 The above cutting method for cutting the single layer winded carbon nanotube is incapable of cutting, at a specified site, the single layer winded carbon nanotube. Namely, the above cutting method is unable to specify the cutting site of the single layer winded carbon nanotube. Since the above cutting method is the chemical wet process using the acid such as  
15 the nitric acid and the sulfuric acid, the acid provides undesirable influence to the manufacturing process. The above conventional cutting method is unsuitable for forming the micro device.

In the above circumstances, it had been required to develop a novel method of processing a nanotube free from the above problem.

20

## SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a novel method of processing a nanotube free from the above problems.

Pf-2727/nec/us/mh

It is a further object of the present invention to provide a novel method of processing a nanotube without using any chemical wet process.

It is a still further object of the present invention to provide a novel method of selectively processing a top portion of a nanotube in a  
5 specific shape.

It is yet a further object of the present invention to provide a novel method of selectively processing a top portion of a nanotube in a specific shape which is suitable for an electron device such as a field emission electron gun.

10 The present invention provides a method of processing a nanotube, comprising the steps of : causing a selective solid state reaction between a selected part of a nanotube and a reactive substance to have the selected part only become a reaction product ; and separating the nanotube from the reaction product to define an end of the nanotube.

15 The above and other objects, features and advantages of the present invention will be apparent from the following descriptions.

## BRIEF DESCRIPTION OF THE DRAWINGS

20 Preferred embodiments according to the present invention will be described in detail with reference to the accompanying drawings.

FIGS. 1A through 1C are fragmentary cross sectional elevation views illustrative of a novel method of forming a top of the single layer winded carbon nanotube in a preferred embodiment in accordance with the

Pf-2727/nec/us/mh

present invention.

FIG. 2 is a view of an image of the top portions of the single layer winded carbon nanotubes taken by the transmission electron microscope in a preferred embodiment in accordance with the present invention.

### DISCLOSURE OF THE INVENTION

The present invention provides a method of processing a nanotube, comprising the steps of : causing a selective solid state reaction between a selected part of a nanotube and a reactive substance to have the selected part only become a reaction product ; and separating the nanotube from the reaction product to define an end of the nanotube.

It is preferable that the step of causing a selective solid-state reaction further comprises the steps of : selectively contacting the part of the nanotube with the reactive substance ; and causing the selective solid state reaction on a contacting region of the selected part of the nanotube and the reactive substance to have the selected part only become the reaction product, wherein a boundary between the reaction product and the nanotube is self-aligned to an edge portion of the contacting region of the selected part of the nanotube and the reactive substance.

It is further preferable that the solid state reaction is caused by heating the reactive substance.

It is further more preferable that the reactive substance is heated

Pf-2727/nec/us/mh

by an irradiation of a heat ray onto the reactive substance.

It is still further preferable that the heat ray is an infrared ray.

It is also preferable that the reactive substance is heated by applying a current between the reactive substance and the nanotube.

- 5 It is also preferable that the step of contacting the part of the nanotube with the reactive substance further comprises the steps of : dispersing a carbon nanotube into an organic solvent to form a dispersion liquid ; applying the dispersion liquid onto a surface of the reactive substance ; and evaporating the organic solvent from the dispersion liquid
- 10 to leave the nanotube on the reactive substance.

It is also preferable that the nanotube is separated from the reaction product by rapidly cooling the reaction product.

It is also preferable that the nanotube is a single-layer winded nanotube.

- 15 It is also preferable that the nanotube is a multi-layer winded nanotube.

It is also preferable that the nanotube is a carbon nanotube.

It is also preferable that the nanotube is a boron nitride based nanotube.

- 20 It is also preferable that the reactive substance is a metal.

It is further preferable that the reactive substance is Nb.

It is also preferable that the reactive substance is a semiconductor.

It is further preferable that the reactive substance is Si.

Pf-2727/nec/us/mh

It is also preferable that the reactive substance is in a solid state.

It is further preferable that the reactive substance comprises a substrate having an edge.

It is further more preferable that the edge is defined by a hole  
5 formed in the substrate.

It is also preferable that the end of the nanotube is a top of the nanotube.

The second present invention provides a method of forming a top of a carbon nanotube, comprising the steps of : selectively contacting a  
10 selected part of a nanotube with a solid state reactive substance having an edge ; carrying out a heat treatment to the solid state reactive substance to cause a selective solid state reaction on a contacting region of the selected part of the nanotube and the solid state reactive substance to have the selected part only become a reaction product, wherein a boundary between  
15 the reaction product and the nanotube is self-aligned to the edge of the solid state reactive substance ; and separating the nanotube from the reaction product to define a top of the nanotube.

It is also preferable that the solid state reactive substance is heated by an irradiation of a heat ray onto the solid state reactive substance.

It is further preferable that the heat ray is an infrared ray.  
20

It is also preferable that the solid state reactive substance is heated by applying a current between the solid state reactive substance and the nanotube.

It is also preferable that the step of contacting the selected part of

Pf-2727/nec/us/mh

the nanotube with the reactive substance further comprises the steps of :  
dispersing the nanotube into an organic solvent to form a dispersion liquid ;  
applying the dispersion liquid onto a surface of the solid state reactive  
substance ; and evaporating the organic solvent from the dispersion liquid  
5 to leave the nanotube on the solid state reactive substance.

It is also preferable that the nanotube is separated from the  
reaction product by rapidly cooling the reaction product.

It is also preferable that the nanotube is a single-layer winded  
nanotube.

10 It is also preferable that the nanotube is a multi-layer winded  
nanotube.

It is also preferable that the nanotube is a carbon nanotube.

It is also preferable that the nanotube is a boron nitride based  
nanotube.

15 It is also preferable that the solid state reactive substance is a  
metal.

It is also preferable that the solid state reactive substance is Nb.

It is also preferable that the solid state reactive substance is a  
semiconductor.

20 It is also preferable that the solid state reactive substance is Si.

### PREFERRED EMBODIMENT

A first embodiment according to the present invention will be



Pf-2727/nec/us/mh

described in detail with reference to the drawings. The present invention is applied to process a top portion of a single layer winded carbon nanotube. The single layer winded carbon nanotube is cut by use of Nb as a first substance to form a top of the single layer winded carbon nanotube. FIGS. 5 1A through 1C are fragmentary cross sectional elevation views illustrative of a novel method of forming a top of the single layer winded carbon nanotube in a preferred embodiment in accordance with the present invention.

With reference to FIG. 1A, a metal substrate, for example, a thin 10 Nb substrate 1 was prepared as a substance which reacts with the single layer winded carbon nanotube. The Nb substrate 1 has a thickness of 2 millimeters. A hole 1a having a predetermined shape was formed by a predetermined position of the Nb substrate 1 by use of an ion milling method. Further, single layer winded carbon nanotubes 2 were prepared by 15 a laser ablation method.

The single layer winded carbon nanotubes 2 were dispersed by an ultrasonic wave into an organic solvent such as ethanol, 2-propanol and acetone to form a single layer winded carbon nanotube dispersed liquid. This dispersed liquid is applied on an upper surface of the Nb substrate 1. 20 An organic solvent is evaporated from the dispersed liquid. In the organic solvent, almost of the single layer winded carbon nanotubes 2 extend as single straight lines or are curved or form a bundle. Namely, the single layer winded carbon nanotubes 2 form a three-dimensional structure in the organic solvent. After the organic solvent was evaporated, then the single

Pf-2727/nec/us/mh

layer winded carbon nanotubes 2 are kept to form the three-dimensional structure over the Nb substrate 1. The single layer winded carbon nanotubes 2 are in contact partially with the Nb substrate 1 at the edge of the hole 1a.

5           The Nb substrate 1 with the single layer winded carbon nanotubes 2 was placed on a heat stage in a vacuum chamber of a ultra high vacuum transmission electron microscope (UHV-TEM, JEM-2000FXVII). The vacuum chamber was sealed and vacuumed to reach a vacuum degree in the range of  $1.33\text{E-}7$  Pa to  $1.33\text{E-}6$  Pa ( $1\text{E-}9$  Torr to  $1\text{E-}8$  Torr) It is  
10 possible that the vacuum chamber is filled with an inert gas such as Ar gas or  $\text{N}_2$  gas at a low pressure to form an inert gas atmosphere in the vacuum chamber.

          With reference to FIG. 1B, this vacuum was still kept and an infrared ray lamp was used to irradiate an infrared ray onto the Nb substrate  
15 1 to heat the Nb substrate 1 up to a temperature which is sufficiently high for causing a solid state reaction between the Nb substrate 1 and the single layer winded carbon nanotubes 2. For example, the highest temperature of the Nb substrate 1 may be in the range of  $800\text{-}1000^\circ\text{C}$ . The heat time may be in the range of 50-60 minutes.

20           The solid state reaction appeared on the contact region between the Nb substrate 1 and the single layer winded carbon nanotubes 2, whereby the contact portion of the single layer winded carbon nanotubes 2 contacting with the Nb substrate 1 became a reaction product 3 of NbC, wherein a boundary between the NbC reaction product 3 and the remaining

Pf-2727/nec/us/mh

single layer winded carbon nanotubes 2 was aligned to the edge of the hole 1a of the Nb substrate 1.

With reference to FIG. 1C, the irradiation of the infrared ray lamp was discontinued and subsequently the sample was rapidly cooled, whereby a crack was formed at the boundary between the NbC reaction product 3 and the remaining single layer winded carbon nanotubes 2, and thus the single layer winded carbon nanotubes 2 were separated from the NbC reaction product 3. As a result, the tops of the single layer winded carbon nanotubes 2 were formed.

FIG. 2 is a view of an image of the top portions of the single layer winded carbon nanotubes 2 taken by the transmission electron microscope. From FIG. 2, it can be understood that the top portions of the single layer winded carbon nanotubes 2 were selectively formed.

In accordance with the novel method of processing the nanotube of the present invention, the solid state reaction is caused on the contact regions of the single layer winded carbon nanotubes 2 contacting with the Nb substrate 1 to cause the contact regions of the single layer winded carbon nanotubes 2 on the Nb substrate 1 become the NbC reaction product, whereby the boundary between the NbC reaction product and the single layer winded carbon nanotubes 2 is aligned to the edge of the Nb substrate 1. The boundary of the single layer winded carbon nanotubes 2 from the NbC reaction product define the tops of the single layer winded carbon nanotubes 2. Non-contact portions of the single layer winded carbon nanotubes 2 structurally remain unchanged. Namely, the tops of the single

Pf-2727/nec/us/mh

layer winded carbon nanotubes 2 are highly accurately formed without changing the structures of the single layer winded carbon nanotubes 2.

The above preferred embodiment may be modified as follows. In the above embodiment, the Nb substrate 1 was used as the substance which reacts with the single layer winded carbon nanotubes 2. Other metals than Nb and semiconductors such as silicon may be available as the substance which reacts with the single layer winded carbon nanotubes 2.

In the above embodiment, the single layer winded carbon nanotubes 2 were used as the nanotubes. Other nanotubes such as boron nitride based nanotubes are also available.

Not only a single layer winded nanotube but also multilayers winded nanotube are available.

In place of the infrared ray irradiation, other heating methods such as a resistance heating method of applying a current between the single layer winded carbon nanotubes 2 and the Nb substrate 1a may be available.

Whereas modifications of the present invention will be apparent to a person having ordinary skill in the art, to which the invention pertains, it is to be understood that embodiments as shown and described by way of illustrations are by no means intended to be considered in a limiting sense. Accordingly, it is to be intended to cover by claims all modifications which fall within the spirit and scope of the present invention.